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A STUDY OF THE ADHESION AND COHESION OF METALS

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# A STUDY OF THE ADHESION AND COHESION OF METALS

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## SUMMARY

In the third quarter of this program, essential modifications of the adhesion testing equipment were designed and constructed to enable the final test schedule to be carried out. Principally, the modifications consisted in the development of a double ion gun bombardment system for impingement cleaning of two surfaces simultaneously prior to contact under load.

In addition, a rapid action specimen selector mechanism was designed to enable rapid positioning of each specimen in the horizontal bombardment and vertical loading orientations. The equipment modifications were expected to be completed by January 31, 1965, at which time the final test series would be conducted in the last quarter of the program.

## INTRODUCTION

In the present research program, National Research Corporation is continuing an investigation of various aspects of solid adhesion phenomena to acquire a deeper understanding of the bonding mechanism. In previous phases of this work, as reported in Quarterly Progress Reports I and II, the effect of material parameters such as mutual solid solubility and contact stress on the adhesion strength was studied.

In addition, a quantitative technique was developed to relate relative changes in the surface electron work function as determined by retarded field diode measurements to the fractional concentration of impurity atoms on surface lattice sites.

Using this method, the degree of oxygen desorption from the target surface after varying period of xenon or argon ion bombardment can be estimated. The relative cleanliness index can then be correlated to the bonding strength.

In the current phase of the investigation, the effect of contact surface profile on the adhesion mechanism is planned for examination. In order to accomplish this, it was necessary to extensively modify the existing adhesion testing apparatus. The main vacuum chamber was redesigned to accomodate a twin ion gun assembly with which it is planned to bombard both contact surfaces simultaneously.

In addition, a rapid action specimen selector mechanism was designed for the loading assembly. With this component, each of the eight sample pairs can be horizontally positioned for xenon ion bombardment. The surfaces selected for test can then be rapidly rotated to the vertical position for contact load application. The entire positioning procedure between cleaning and contact should require less than ten seconds.

To complete the design, construction and assembly of the new components, it was anticipated that the third quarter period of the program would be required. Accordingly, permission was requested of the sponsoring agency to extend the calendar schedule of the program. This report will then briefly cover the design modifications completed during the reporting period. Experimental tests are scheduled for the fourth quarter extension period.

## EXPERIMENTAL APPARATUS

### General Design

The adhesion testing apparatus used in the present work is shown in Fig. 1. It consists of a stainless steel, cylindrical ultra-high vacuum chamber, 14 in. in diameter, an axial loading mechanism for adhesion specimens, several ports for the introduction of surface cleaning components and vacuum gauges together with the associated electrical and mechanical controls.

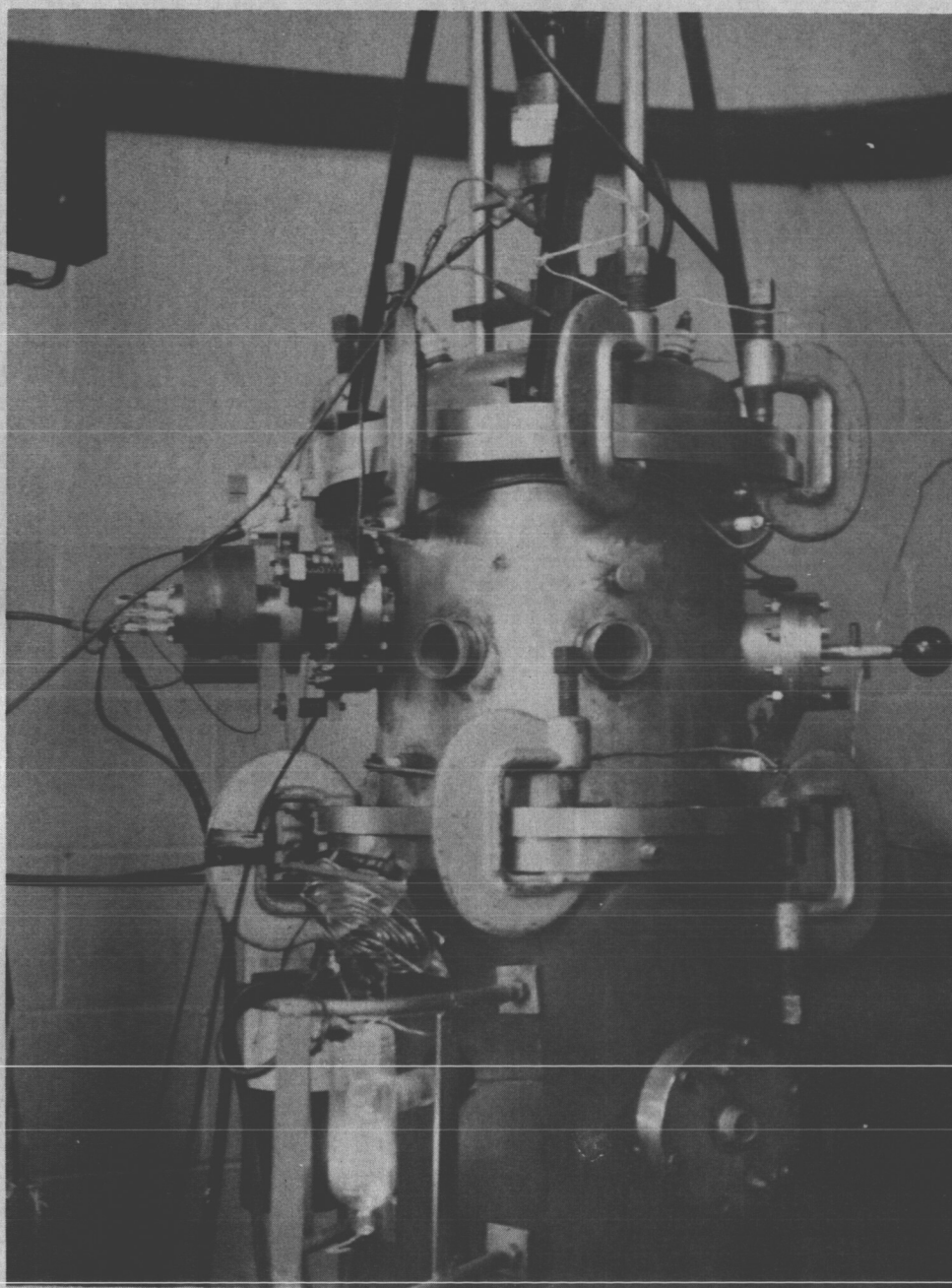


Fig. 1

ADHESION TESTING APPARATUS



The vacuum chamber is evacuated by means of a 10 in. NRC diffusion pump backed up by a smaller 2 in. diffusion pump and a mechanical pump. The exit orifice passes through a 90° bend with an internal liquid nitrogen trap to reduce the possibility of diffusion pump oil influx. Key flange seals are refrigerated or water cooled to prevent out-gassing of the "o"-ring seals. The system can be heated to 250°C using internally mounted quartz rod heaters for thermal outgassing. The vacuum system is capable of maintaining pressures below  $1 \times 10^{-9}$  torr under test conditions.

An integral part of the test equipment is the loading mechanism, illustrated in Fig. 2. The upper and lower loading stages comprise disc-shaped multi-specimen holders which can sequentially select the adhesion surfaces for test. In this way, up to eight adhesion tests may be performed during the course of one pumping operation. The upper loading shaft contains a vacuum sealed ball and screw drive mechanism which can impose loads up to 2200 pounds on the lower anvil. The force is monitored by a load cell and is applied by a motor driven chain sprocket mounted outside the vacuum chamber. A rigid compression cage fixture is provided to guide the motion of the upper shaft and ensure vertical alignment of the loading stages.

The vacuum assembly also contains a wire brush abrasion device for scouring contaminant films from the adhesion surfaces immediately prior to contact. The cylindrical brush is mounted on a flexible bellows seal to permit manual manipulation from outside the chamber. Rotary power is provided by a compact vacuum sealed motor integrally mounted on the brush shaft. In practice, the brush, usually of stainless steel, is brought to bear on the two test surfaces simultaneously, as shown in Fig. 3. After a prescribed abrasion period, the brush is swung away and the surfaces are brought into contact under load.

## DESIGN MODIFICATIONS

### Ion Gun Assembly

Since the brush abrasion technique necessarily roughens the surface severely, it was considered desirable to devise an inert gas ion beam cleaning method for both contact surfaces in examining the effect of surface profile on adhesion





Fig. 3

WIRE BRUSH ABRASION DEVICE

strength. Because the mating surfaces cannot be brought close together in the test apparatus except in the parallel position, two separate ion gun assemblies were required to bombard each surface independently. In order to accomplish this in the limited space available in the vacuum chamber, it was planned to mount the two guns on the side of the cylindrical chamber at positions  $90^\circ$  apart. In this way, the ion beams could be focused independently on the specimen surfaces when rotated to the horizontal position in the loading mechanism.

An individual gun assembly is shown in Fig. 4. Consisting essentially of a cold cathode ion source based on the Redhead cold cathode magnetron gage which can ionize a partial pressure of xenon or argon gas bled into the central cavity, the gun can direct an energized stream of ions to the target surface by means of a series of einzel lenses. The magnet components are mounted outside the gun cavity to permit removal during thermal outgassing. The ion stream is directed into the vacuum chamber through a narrow orifice to minimize pressure build-up in the main chamber.

In practice, anode voltages up to 4000 volts at  $1 \times 10^{-6}$  amp can be consistently obtained, yielding ion current densities up to 12 amp/torr -  $\text{cm}^2$  at the target surface. In the usual operating condition, the partial pressure of inert gas in the gun cavity may be  $10^{-2}$  torr; the corresponding system pressure will rise to  $2 - 5 \times 10^{-7}$  torr during bombardment. In use, the ion stream may be deflected by manual operation of a bellows seal spring assembly thus permitting accurate focusing of the beam on the target. Focusing is accomplished by monitoring the optimum current peak during the deflection adjustment.

The installation of the gun assemblies in the vacuum chamber is shown in Fig. 5. The radial magnet components are shown in position. Each gun assembly is provided with a Granville-Phillips bleeder valve which admits a controlled quantity of high purity xenon gas to the gun cavity through a 0.040 in. inlet tube. The deflection spring coils providing angular movement of the gun axis at the flexible bellows seal are also shown.

A third inlet to the chamber provides for operation of the wire brush abrasion device through a flexible bellows seal. Additional flexible manipulators are provided for operation of the loading mechanism. Lastly, several viewports are present for visual observation of the adhesion apparatus during test.

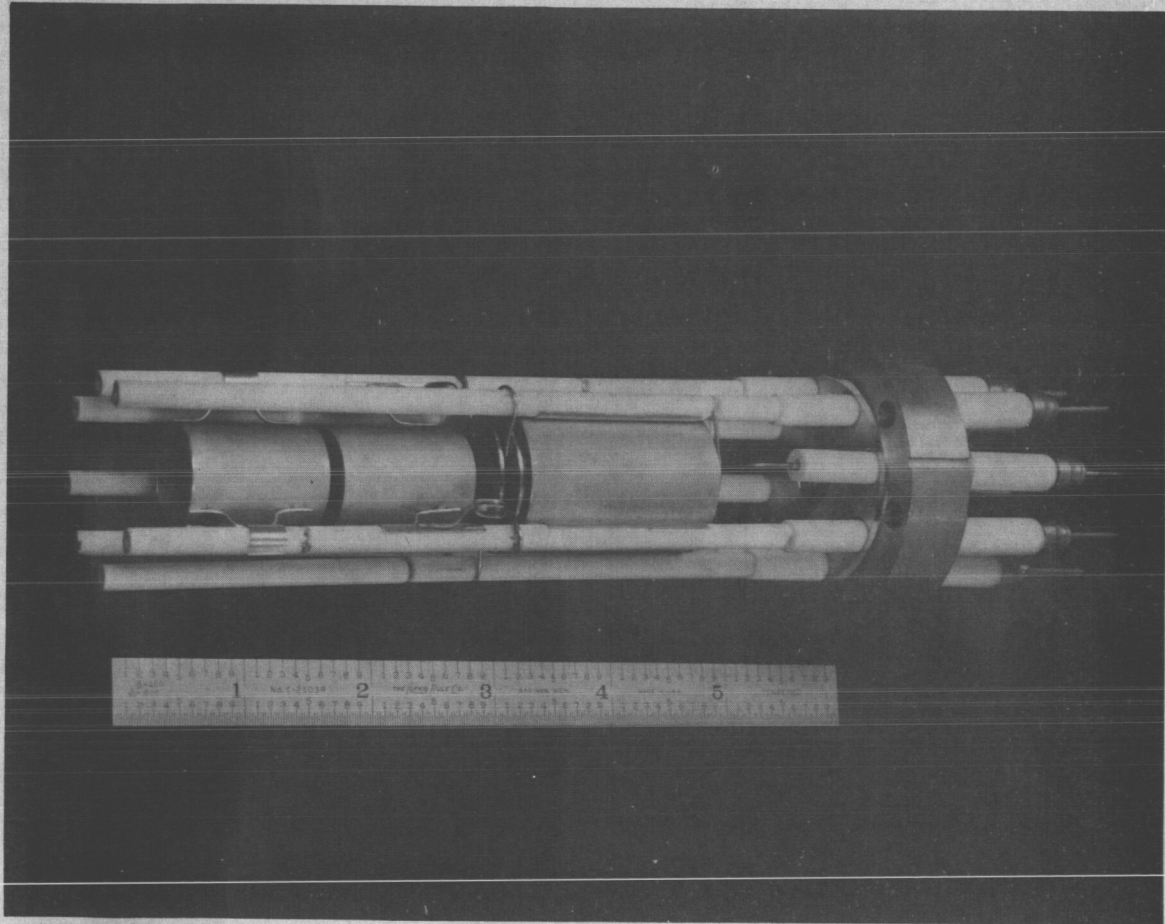


Fig. 4  
ION BEAM GUN ASSEMBLY



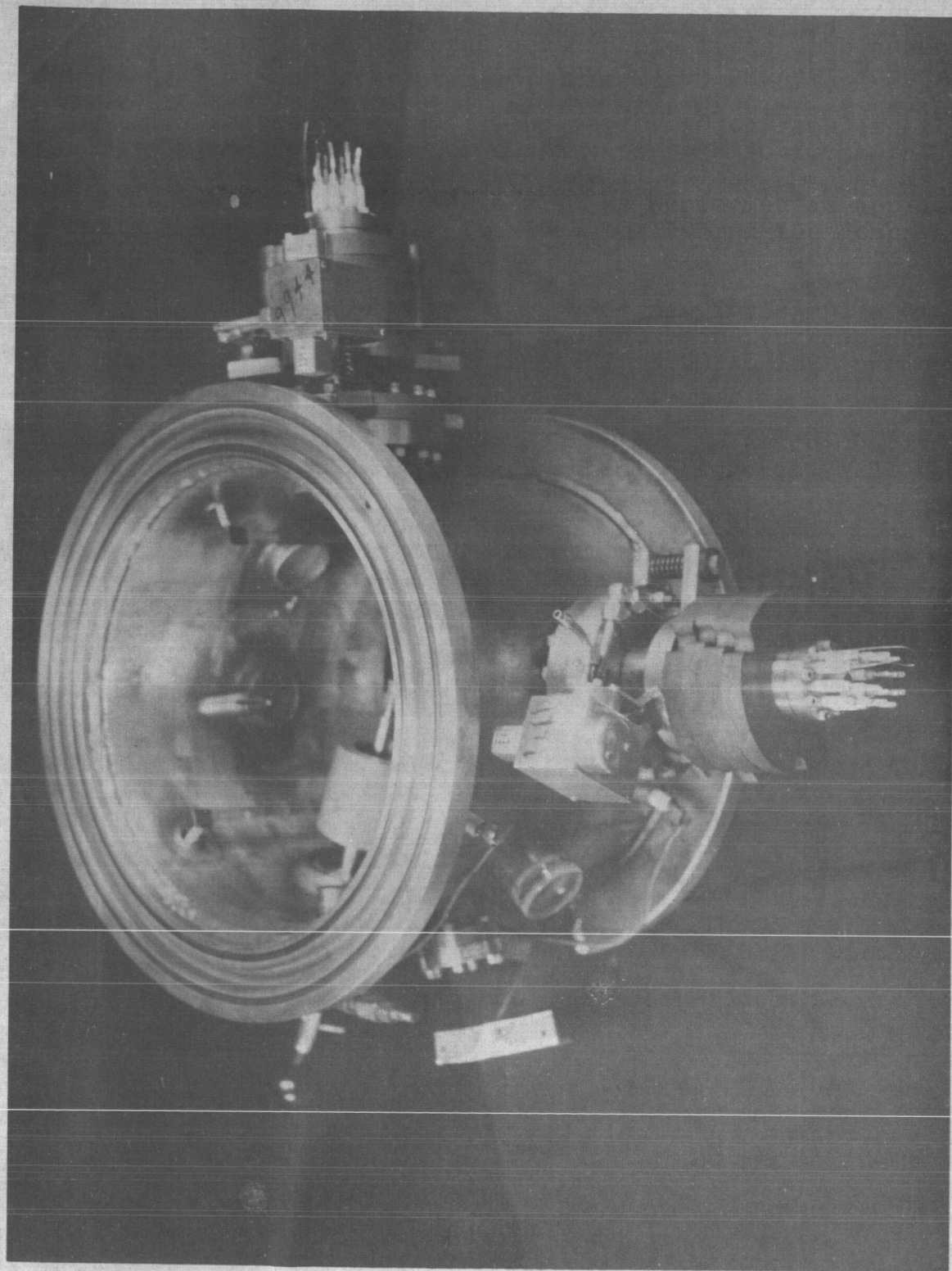


Fig. 5

INSTALLATION OF TWO ION GUNS AND WIRE BRUSH DEVICE IN ADHESION TEST CHAMBER



## Loading Mechanism

Due to the requirement that the specimen surfaces be brought to a horizontal position for ion bombardment and then to a vertical position for loading, rapid rotation of selected surfaces through  $90^\circ$  is necessary to minimize the possibility of oxide film reformation after cleaning.

In the original design, the upper and lower indexing heads containing eight specimen stations could only be rotated in one direction, in a station-to-station sequence. With each rotation of  $45^\circ$ , the indexing heads were locked by a spring-loaded pin, preventing further motion during the loading sequence. Rotation through  $90^\circ$  thus required two manipulative steps and the expenditure of up to 40 sec. in total positioning time.

To reduce the time required for rotation, the indexing heads were modified by the addition of a spring-loaded negative-torque drive as illustrated in Fig. 6. In this arrangement, unlocking the set pin by manipulative action permits rapid rotation of any one of the stations to the desired position with a consequent reduction in positioning time to less than 8 sec. Dependent to some degree on operator skill, the elapsed time between bombardment cleaning and actual contact can now be reduced to about 12 sec. In comparison, the elapsed time in wire brush cleaning averaged about 18 sec.

## EXPERIMENTAL PROGRESS

During the reporting period, the design modifications outlined in the previous section have been largely completed. Preliminary tests are currently underway on the performance of the ion guns and specimen selector mechanism. In operation, it has proved feasible to supply both guns from a single xenon source. However, the gun assemblies have independent high voltage power supplies and electrometers.

Experimental tests on adhesion samples of varying surface profile are scheduled for the last quarter of the program.

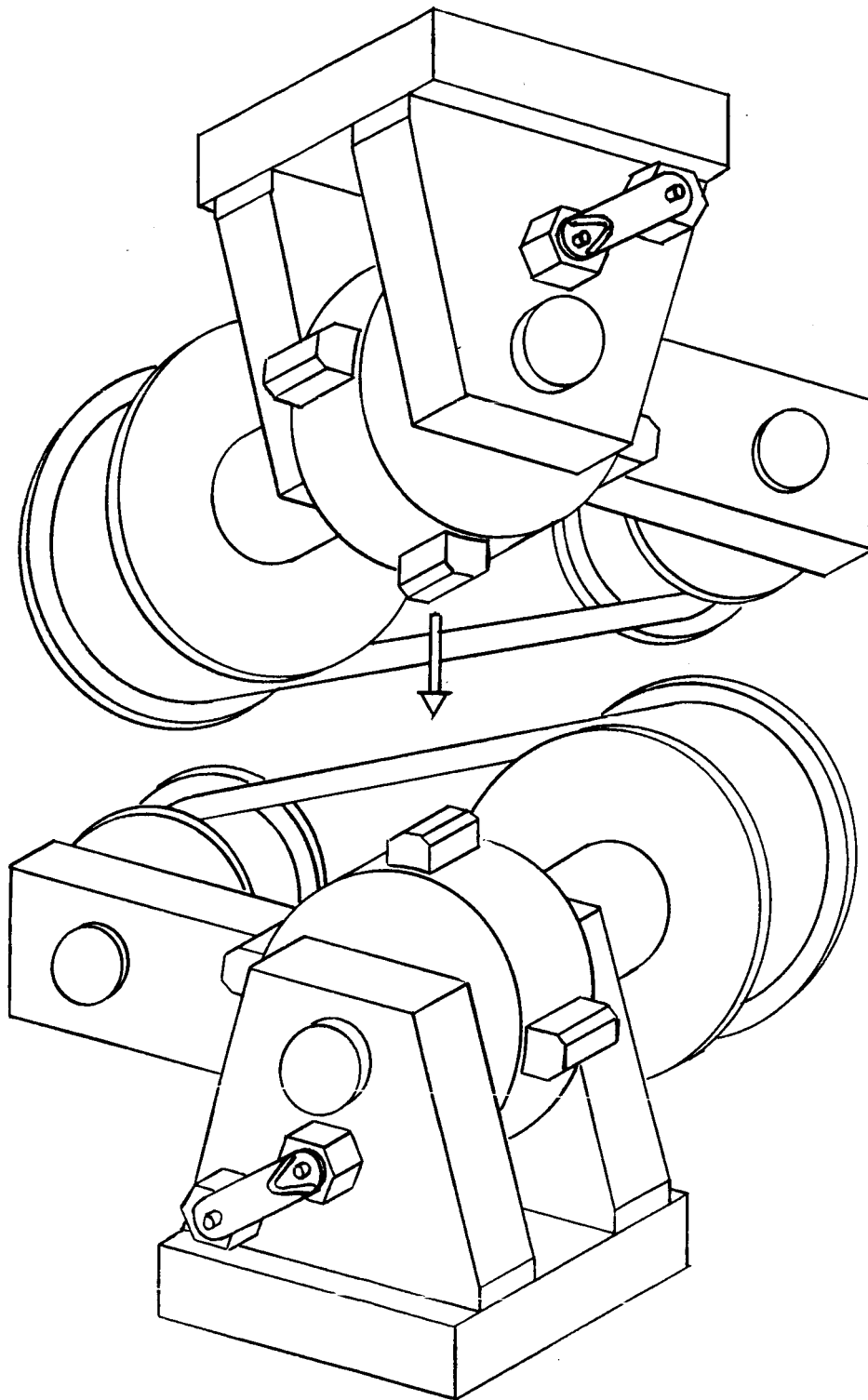


Fig. 6

DETAILED ASSEMBLY OF MODIFIED SPECIMEN SELECTOR AND LOAD MECHANISM

## FUTURE WORK

Upon completion of the performance tests, the adhesion apparatus will be utilized for the following test schedule:

1. Adhesion tests will be performed on samples of copper with two degrees of surface preparation; smooth-polished surfaces with average profiles less than 4 rms height variation. The tests will be performed at applied contact stresses ranging from 50% to 150% of the compressive yield stress of copper.

2. Similar tests will be performed for samples of gold and iron in both the polished and roughened surface conditions at applied stresses ranging from 50 to 150° per cent of the yield strength of interest.

3. Surface profile measurements (rms height variations), ion beam bombardment data, and adhesion strength will be determined for each material and condition.